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EFFECT OF ELECTROLYTE WATER CONTENT ON THE ANODIC
PASSIVATION OF LITHIUM I.. (U) NAVAL SURFACE WEAPONS
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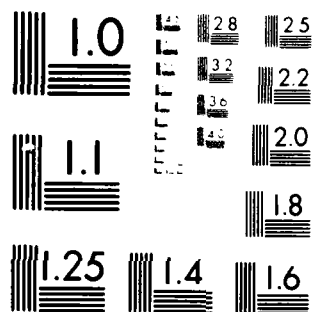
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**EFFECT OF ELECTROLYTE WATER CONTENT ON
THE ANODIC PASSIVATION OF LITHIUM IN
1M LiClO_4 -PROPYLENE CARBONATE**

BY S. D. JAMES,
A. R. NAGAO

RESEARCH AND TECHNOLOGY DEPARTMENT

JUNE 1982

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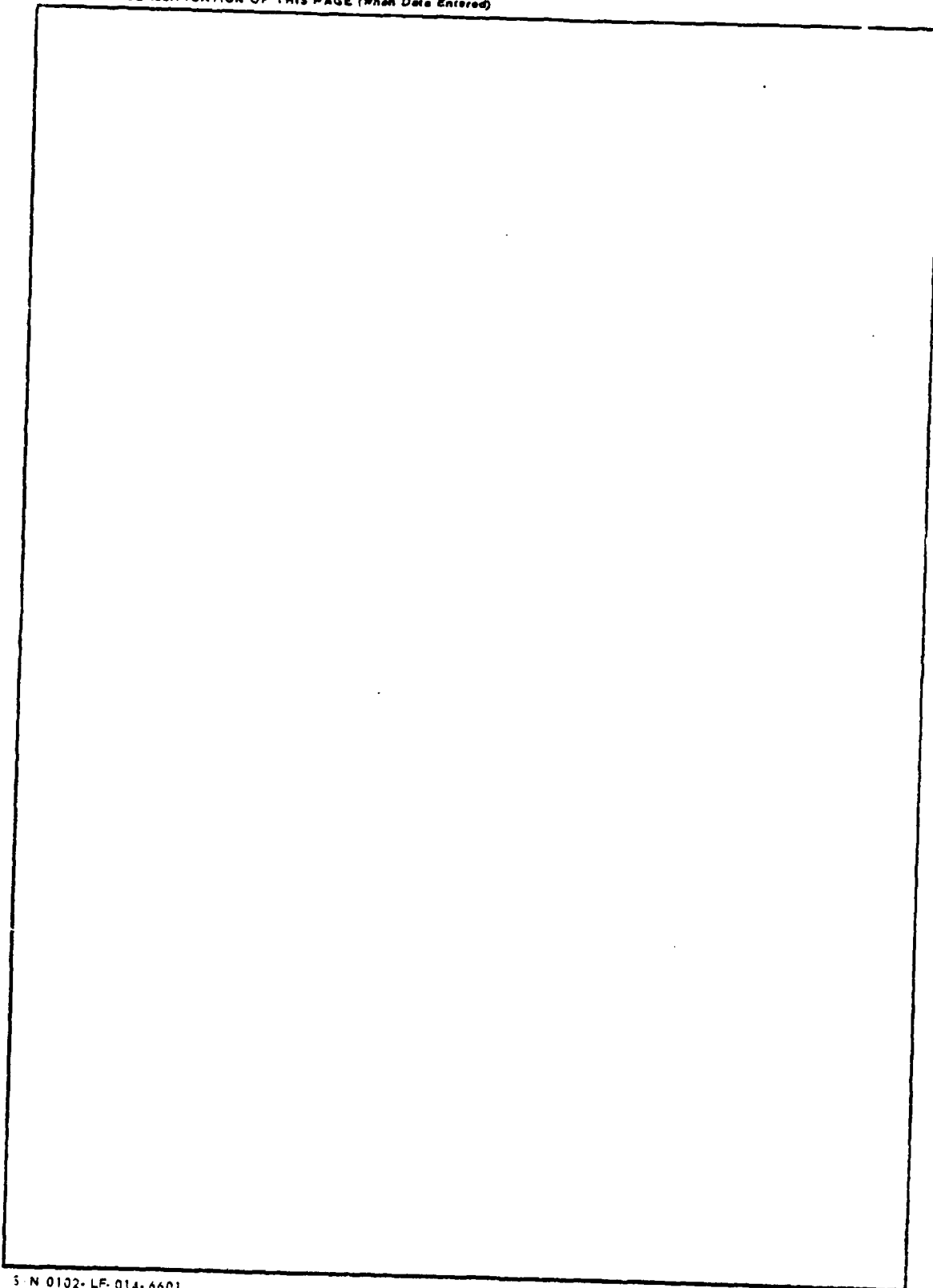
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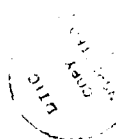
FOREWORD

This report describes the effect of aqueous contamination on the anodic passivation of lithium electrodes in 1M LiClO₄ - propylene carbonate. This work relates to safety problems that have been experienced with anode-limited lithium batteries. We acknowledge the financial support of the Independent Research Program of the Naval Surface Weapons Center in conjunction with the Electrochemistry Technology Block Program.

Approved by:

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CHAPTER 1

INTRODUCTION

Safety problems in both the Li-SO₂ and the Li-SOCl₂ batteries have delayed their introduction into advanced weapons systems. The present work seeks to elucidate hazards originating at the anode (Li) side of the battery. It has been reported that anode-limited Li-SO₂¹ and Li-SOCl₂² cells can vent or explode when driven into voltage reversal, especially if metallic Li remains at the failed anode. We have recently shown³ that above a certain controlled current density a Li anode suffers eventual anodic passivation. This terminates the anode's useful life and forces its voltage to high positive values where anode substrate or anolyte species are oxidized. Since unconsumed metallic Li remains at the anode after anodic passivation, this event brings about the hazardous conditions mentioned above and thus merits detailed examination. The present report recounts the role of water contamination on the anodic passivation of Li in 1M LiClO₄ propylene carbonate (PC).

¹Dey, A. N., "Safety Studies in Li/SO₂ Cells," J. Electrochem. Soc., 127, 1980, p. 1886.

²Abraham, K. M., Holleck, G. L., and Brummer, S. B., "Studies of Explosion Hazards of Li/SOCl₂ Cells on Forced Overdischarge," Battery Design and Optimization, S. Gross, ed. (Princeton, New Jersey: The Electrochemical Society Softbound Proceeding Series, 1979), p. 356.

³James, S. D., "The Anodic Passivation of Lithium," submitted to J. Electrochem. Soc.

CHAPTER 2

EXPERIMENTAL

1. Test Cell And Discharge Procedure

The electrochemical test cell is made of Pyrex glass and contains about 100 ml of electrolyte under a cover gas of dried argon. Two sizes of test electrode were used: an 8 cm² film of Li smeared over a Stainless Steel (SS) 304 disc and a 0.08 cm² surface Li electrode formed by tamping Li into a 1/8" hole in a SS block. The higher area test electrode is illustrated in Figure 1. Both electrodes faced upwards toward a SS or Al disc counter electrode about 15 mm above. Lithium, plastered over the threaded tip of a 3 mm SS rod, served as reference electrode. Discharge curves were recorded at constant current, anodic to the test electrode at $23 \pm 1^\circ\text{C}$.

2. Desiccant Activation And Electrolyte Desiccation

Two types of solid desiccant were employed to dehydrate solutions. Linde Type X Molecular Sieve was obtained in the lithium form (LiX). It was activated by a series of progressive temperature elevations of roughly 100°C (from room temperature to 500°C) with continuous pumping over a period of five days. Alcoa F-1 alumina was activated by continuous pumping and heating at 270°C for a period of seven days. Electrolytes were desiccated by percolation through an 8" x 18 mm column of desiccant containing Molecular Sieve, alumina or a combination of both. Percolation rates depended on the level of desiccation desired (usually 5-10 seconds/drop corresponding to 20-40 ml/hour). Both column and receiver were of Pyrex glass and their atmospheres were closed and looped together via Viton tubing to exclude the lab atmosphere during these protracted percolations. Percolation was done in the Dry Room or in the Helium glovebox.

3. Determination Of Electrolyte Water Content

This was done using Metrohm's Karl Fischer automatic titrator obtained from Brinkmann Instruments, Cantiague, NY. We used the model E547 Titrator with the EA 875-5 Titration Vessel and the E535 or E415 Dosimats. Working under a positive pressure of slowly flowing dried Ar in the Titration Vessel we were able to reproduce water standards in PC to ± 20 and $\pm 7\%$ at the one and five ppm water levels respectively. However, we found that in this low water content region the apparent water equivalence of the Karl Fischer Reagent was smaller at the lower ppm due to water ingress from the "dry" Ar cover gas. Thus in view of our particular interest in this low ppm region we transferred all our operations (electrolyte desiccation, water titration and passivation studies) to a Vacuum Atmospheres Co, Helium atmosphere glovebox, model # HE-553-2 (water below 0.5ppm). Under these conditions the reproducibility of water titers of our electrolytes was $\pm 20\%$ at the 10 ppm level. This was much inferior to that of the water standards for reasons presently obscure to us.

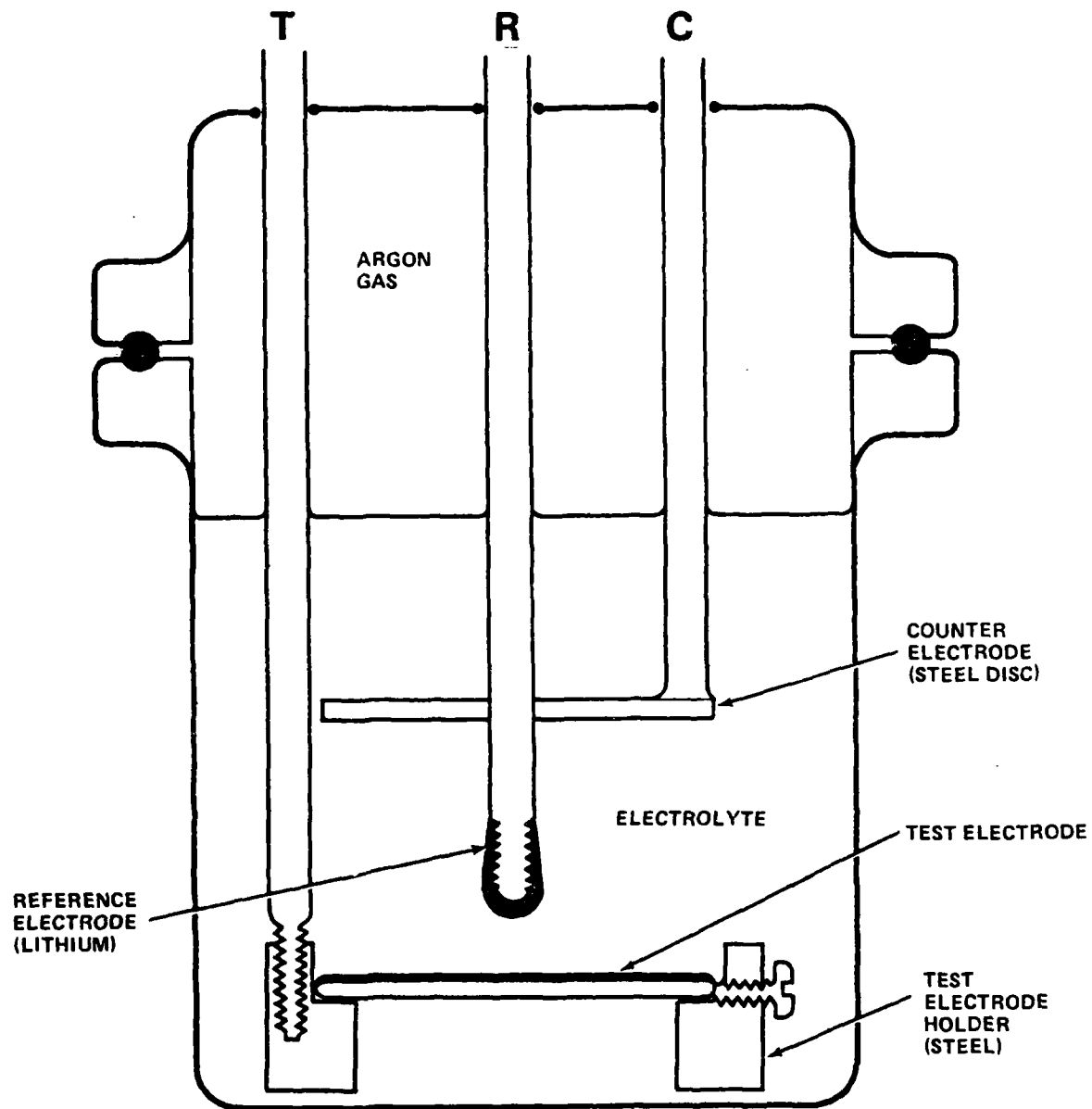


FIGURE 1. DISCHARGE CELL

4. Attempts To Lower Electrolyte Water Content Below 1ppm

(a) By percolation through desiccant columns

It was earlier believed that properly activated desiccants (Molecular Sieve or alumina) would be able to dry electrolytes to below one ppm. However, in $\text{LiClO}_4\text{-PC}$, it was found that a water level of only around five ppm could be reached by this method. In spite of cutting the percolation rate to one drop in twenty-five seconds (about four ml per hour) and multiple passes through the column, water content never fell below about five ppm.

(b) By using a drier salt at lower concentration (0.1M LiAsF_6)

We obtained a very dry grade of LiAsF_6 (Electrochemical Grade from US Steel Corporation). It was nominally 50-100 ppm in water but our analysis showed it to be actually only eight ppm. Thus, 0.1M and 1.0M solutions of this salt in a dry solvent should have only 0.14 and 1.4 ppm water respectively. However, we found that our usual 0.16A (about 2 min passivation time) passivation runs were not feasible in the 0.1M electrolyte. The higher resistance of this less conductive electrolyte generated IR drops causing the steel substrate of the Li anode to anodically dissolve, contaminating the solution with Fe, Cr or Ni ions. So we abandoned this approach. In the future it might be useful to use the more conductive 1.0M solution in runs at 1.4 ppm.

(c) By preelectrolysis

We tried to dry electrolytes by preelectrolysis in the same electrochemical cell used for passivation studies. Current was passed to anodically dissolve the 8 cm^2 lithium test electrode and plate dendritic lithium on the steel counter electrode. We hoped that the active lithium dendrites (high area, free lithium formed by the reduction of lithium ions at the steel cathode surface) would be able to desiccate the electrolyte:



Preelectrolysis was found to be effective in reducing the water ppm of $1.0\text{M LiClO}_4\text{-PC}$ from 105 to 11 after 45 hours of electrolysis at 1 mA/cm^2 (8mA). Electrolysis was done in the helium glovebox with continuous stirring of the electrolyte to maximize contact between dendrites and solution. So we next predried another batch of this electrolyte by percolation through LiX and then subjected it to exhaustive electrolysis to hopefully finish the job. However, in spite of varying conditions of current density and electrolysis time, we were unable to reduce the water content below five ppm. It is possible that this apparent residual water might be due to the inefficient exclusion of lithium dendrites from the electrolyte aliquot pipetted into KF Titration vessel. Some tiny dendrites may have passed the two layers of predried paper tissue wrapped around the plastic tip of the Oxford automatic macropipet.

Due to the frustration of our efforts to dry electrolytes below 1 ppm, we were forced to alter our plan so that, for the time being at least, we would study lithium passivation in the region upwards of 5 ppm water content.

5. Dendrite Control

During passivation runs (as during preelectrolysis) Li dendrites form at the SS counter electrode (cathode) and become suspended in the electrolyte. This became a major problem because firstly the electrolyte's water content will change with time if these active dendrites are continuously being formed and reacting as in equation (1); some method must be devised to contain them. Secondly, the dendrites consume Karl Fischer reagent so that titration gives an exaggerated value for the electrolyte's water content. This problem was solved by using a modified Oxford pipet tip fitted with a porous filter to withdraw the sample from the electrochemical cell. Samples of electrolyte were then presumably filtered dendrite-free.

To circumvent the first problem, enclosure of the counter electrode by a porous separator was necessary. We first tried enclosing the counter electrode in a Pyrex tube terminating in a 20 mm coarse Pyrex frit but unfortunately, its resistance to current flow was too high for our 40 volt power source to compensate for. We then enclosed the counter electrode in a similar Pyrex tube where the frit was replaced by wrapping the end of the tube in 2 layers of predried paper tissue. This worked at first but soon the dendrites started to clog the tissue and the resistance increased to an unacceptable level. The situation is aggravated by lithium's proclivity for growing towards the anode under the influence of the IR drop in the electrolyte (see below). The problem of Li dendrites originating at the counter electrode was eventually solved by:

(a) Using an aluminum instead of a steel disc as counter electrode. At low cathodic current densities, aluminum absorbs deposited lithium as a solid, non-dendritic Li/Al alloy. (b) Drastically reducing (by x 100) the area of the lithium test electrode. This proportionately cuts the current needed to characterize lithium passivation and lowers cathodic current density to values where only solid Li/Al forms at the counter electrode. A new design of test electrode was built to accomplish this. Raising the aluminum counter electrode out of the electrolyte between passivation runs minimizes Li/Al's reaction with water and change of its ppm.

A second source of dendrites took us by surprise. After carefully eliminating the counter-electrode's dendrites we still saw dendrites swirling round the stirred electrolyte after a period of electrolysis. We traced these to a "beard" of dendritic lithium growing on the tip of the lithium reference electrode. Even though this lithium is electrically isolated from the current-carrying circuit it can apparently undergo local cell action whereby its top anodically dissolves, and an equal mass of lithium plates dendritically onto its tip. This is powered by the electric field of the IR drop in the adjacent electrolyte. We solved this one by wrapping the lithium reference in four layers of predried paper tissue and raising it to the top of the electrolyte out of most of the IR drop between test and counter electrodes.

CHAPTER 3

RESULTS AND DISCUSSION

(a) Anodic Passivation Curve

Figure 2 shows a typical example of the anodic passivation of Li at constant current. The first curve in a series of runs sometimes contains the peak labeled "anodic activation" where anodic current is disrupting a passivating film of Li salt responsible for the well known "voltage-delay" effect. Then after a plateau at the Li voltage, anodic passivation occurs and anode voltage rises to a second plateau where the steel substrate and/or anolyte species are oxidized. This voltage transition is not due to the complete consumption of Li because a few minutes wait at open-circuit suffices to depassivate the surface and the process can then be repeated as in Figure 1 to give a fairly reproducible τ .

The product of the current density ($A.cm^{-2}$) and the transition time τ (sec) gives the passivation charge Q ($C.cm^{-2}$) necessary to passivate the Li anode. In unstirred 1M $LiClO_4$ -PC at room temperature, Li does not passivate below about $1m A.cm^{-2}$, i.e., Q is infinite. Q falls with rising current density leveling off at about $2 C.cm^{-2}$ at $100 mA.cm^{-2}$. Thus in this region, τ varies inversely with current as though a discrete film were forming on the Li surface.

(b) Effect Of Electrolyte Water Content On Anodic Passivation

Other known examples of anodic passivation are thought to involve the presence of water which allows films of metal oxide or hydroxide to cover the surface of the anodizing metal. In fact iron does not passivate in non-aqueous solution unless traces of water are added (4). The passivating effect of these films may be exerted in two ways: (a) directly by impeding the flow of electrons and ions across the metal-solution interface and (b) by providing a porous, superficial film of oxide/hydroxide in the pores of which the anodically generated, electrolyte-salt (in our case $LiClO_4$) can accumulate until it precipitates out as an impervious layer. The first question is: does water content play a role in Li's anodic passivation in our system?

⁴Sato, N. and Okamoto, G., "Electrochemical Passivation of Metals," Comprehensive Treatise of Electrochemistry, B. E. Conway, ed., Vol. 4 (New York: Plenum, 1981), p. 193.

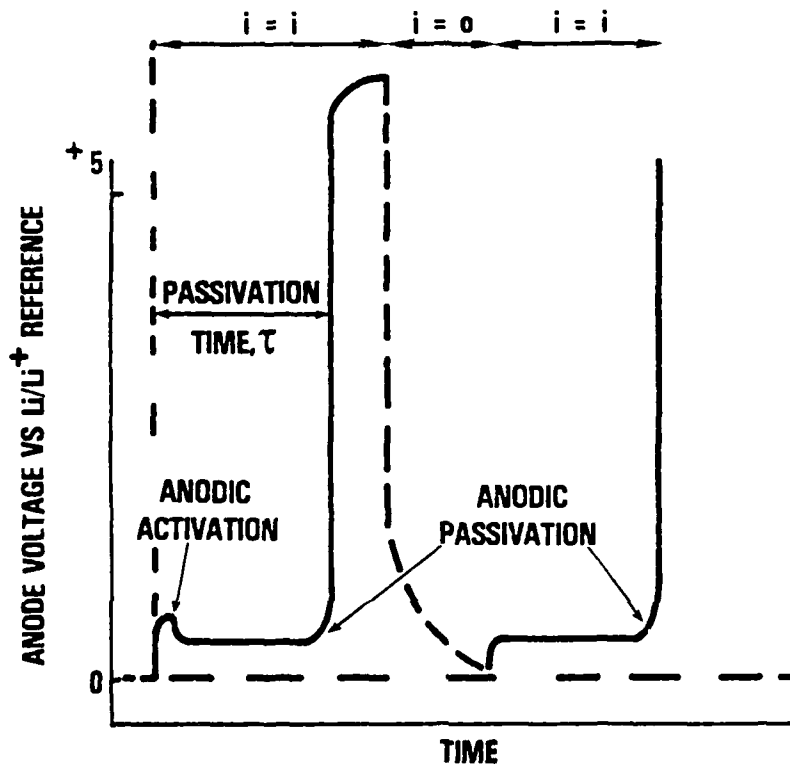


FIGURE 2. ANODIC PASSIVATION OF LITHIUM AT CONSTANT CURRENT

Figure 3 answers a guarded yes to that question. It shows transition time τ for Li passivation versus electrolyte water content. Each τ is the average of three to five determinations. Curve B was actually obtained first. It resulted from a series of experiments over a period of about two months with a number of different 8 cm² Li electrodes and different electrolyte samples under flowing predried argon gas. Despite the large scatter in this data it is reasonable to draw curve B showing a general downward trend, i.e., Li is more easily passivated in the moister solutions. The two crosses in curve B refer to solutions that had been subjected to protracted preelectrolysis. While the intent of this treatment was dehydration, another effect would be to remove heavy metal ions from solution. Since these points lie on the same curve as the others, we can exclude heavy metal ion contamination as being an important factor in the anodic passivation. Curve A, on the other hand, was obtained in the same cell over a period of two hours using a small (0.08 cm²) Li electrode and working in the He glovebox. The electrolyte was first desiccated to 15 ppm by percolation through LiX and the average τ was measured. Then successive water additions were made via a microsyringe, water content checked by Karl Fischer titration and average τ redetermined. Again, there is a clear fall in τ with increasing water content though small relative to that in curve B. The reason for this smaller effect is not clear to us. It remains possible that a very much larger τ /ppm effect occurs below 5 ppm, a region inaccessible to our limited desiccating ability.

An apparent anomaly in Figure 3 is the fact that the τ 's of curve A, at 35 mA.cm⁻² substantially exceed those of curve B at 20 mA.cm⁻² whereas the reverse would be expected. This is because both the 8 cm² and the 0.08 cm² electrodes were unshielded, i.e., the Li was not recessed to enforce perpendicular diffusion to the Li surface. Thus edge diffusion occurred at both electrodes and was much more significant at the smaller electrode due to its ten times higher edge/surface ratio. This is an indication of the importance of mass transport in the anodic passivation process.

(c) Effect of Chloride Ion on Lithium Passivation

Both chemical and anodic passivation are well-known with metals like Fe, Cr and Ni and various steels. Chloride ion is very effective in breaking down passivity and promoting the corrosion of such metals due probably to its strong specific adsorption on their surfaces. We wished to compare its behavior in the case of lithium. So we first measured Q (anodic charge needed to passivate lithium) in 1.0M LiClO₄-PC at 20 mA/cm² as 3.92 ± 0.12 coul/cm². Then we saturated this solution with LiCl by stirring about 0.2g of LiCl into the 80 ml of cell solution. Redetermination of Q gave a value of 3.86 ± 0.30 , i.e., passivation was unaffected by the presence of chloride ion. The striking difference between the two systems may be linked with lithium's much more negative electrode potential (by perhaps 2 volts). This would electrically repel the negative chloride ion, preventing its absorption and disabling it for any depassivation effect.

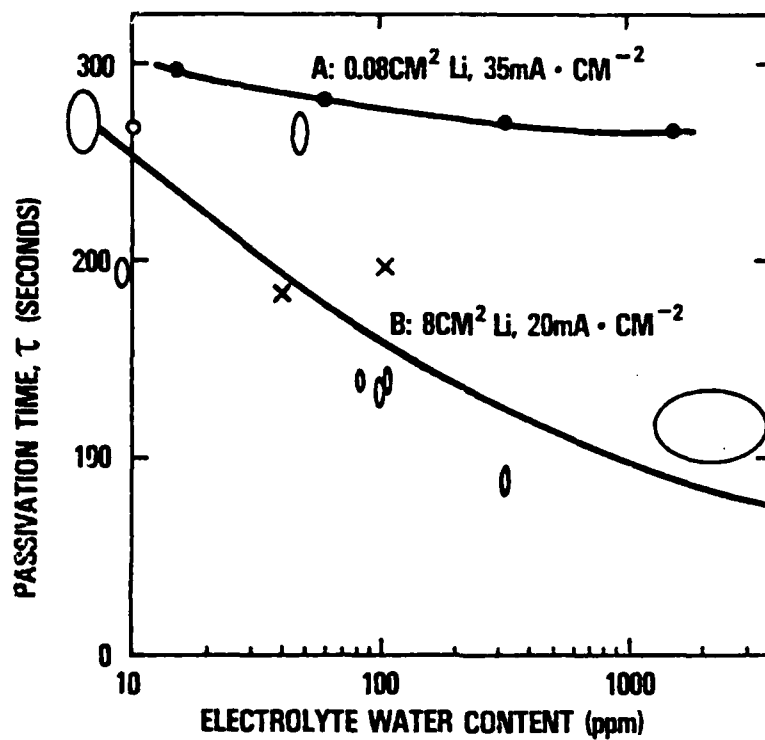


FIGURE 3. ANODIC PASSIVATION TIME VERSUS ELECTROLYTE WATER CONTENT FOR Li IN UNSTIRRED $1\text{M LiClO}_4 - \text{PC}$ AT 23°C .
X: ELECTROLYTE SUBJECT TO PREELECTROLYSIS

(d) Conclusions and Future Work

Despite Figure 3's conflicting evidence as to the size of the effect, the data does clearly indicate that water has a role in the anodic passivation of Li in LiClO_4 -propylene carbonate. The fairly rapid (several minutes) depassivation of the anode at open-circuit shows that the passivating film, whatever its nature, must leave the Li surface rather quickly by mechanical breakdown or by physical/chemical dissolution. The pronounced edge-effect, indicating the role of mass-transport in the passivation process suggests that the precipitation of anodically generated LiClO_4 may cause passivation. Water may cooperate by filming the Li with a porous layer of lithium oxide/hydroxide which retains LiClO_4 close to the Li surface.

Future work should employ improved drying techniques to explore the region below 5 ppm water content. Also we should evaluate the effect of different anions and solvents in the electrolyte under controlled conditions of water content. Review of this data should then allow more definitive conclusions as to the nature of the passivation process and recommendations for avoiding this event in lithium battery use.

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